THE DETERMINATION OF POTASSIUM IN CEMENT WITH TETRAPHENYL BORON

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Joint Highway Research Project

by

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Final Report

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TO: K. B. Woods, Director
Joint Highway Research Project

July 1, 1959

FROM: H. L. Michael, Assistant Director
Joint Highway Research Project

File: 4-6-7
Project: C-36-476

Attached is a report entitled, "The Determination of Potassium in Cement with Tetraphenylboron", by Dr. W. L. Dolch of our staff.

This short article reports the finding of a direct and simple method for the determination of potassium in portland cement. It is presented to the Board for the record.

Respectfully submitted,

H. L. Michael

H. L. Michael, Assistant Director
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HLM:lmc

Attachment

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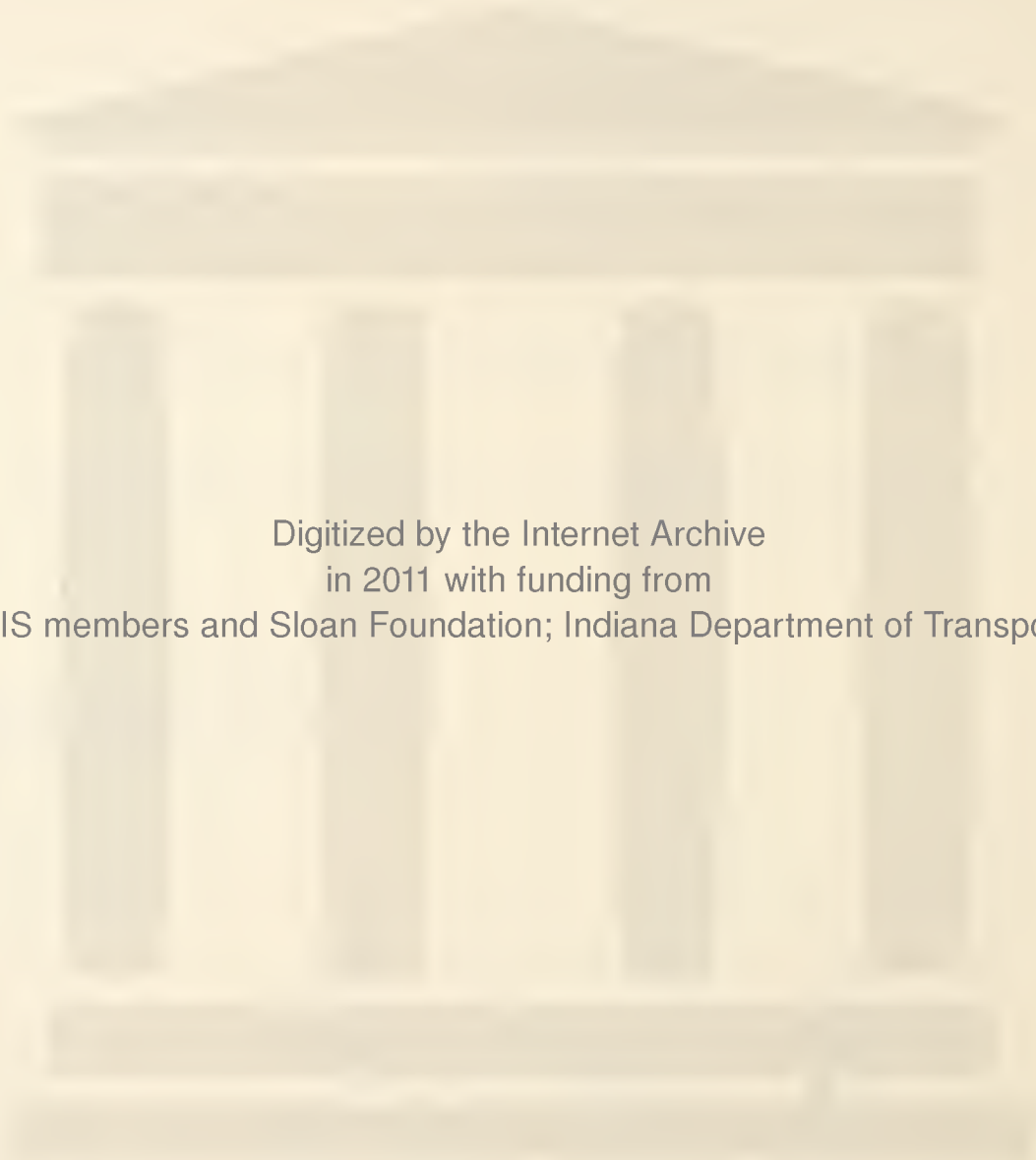

Final Report

THE DETERMINATION OF POTASSIUM IN CEMENT WITH TETRAPHENYLBORON

by

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Joint Highway Research Project
File 4-C-7
Project C-36-7a
476

Purdue University
Lafayette, Indiana
July 1959

The Determination of Potassium in Cement with Tetraphenylboron

Introduction

The two conventional methods for the determination of potassium in portland cement are the chloroplatinate precipitation and the flame-photometric determination. These methods are recognized by the A.S.T.M. in their standard procedures (1, 2).*

Each of these methods, has, however, certain disadvantages. The chloroplatinate precipitation, long the standard reference method, is a difficult analysis to perform properly and requires both an expensive reagent and a comparatively long time. The flame-photometric determination, while excellent for the routine analysis of many samples, requires an expensive instrument and frequent calibration and is uneconomical for the laboratory that is called upon only for a relatively infrequent determination of this component. There is a need, therefore for an easy, rapid, and inexpensive method for this analysis.

In recent years the use of sodium tetraphenylboron as a precipitant for potassium ion has gained considerable popularity. A comprehensive bibliography on the properties and uses of this reagent has been prpared by Barnard (3). It has been shown that this reagent is a quantitative precipitant for potassium and that the pH of the solution should be kept low to give rise to an easily-filtered precipitate that is free from interference by co-precipitation of other metals (4). On the other hand, precipitation in highly acidic solution may

*Number in parthenceos refer to references

cause decomposition of the reagent and consequent high results and this can be prevented by cooling the solution to around 0°C before the precipitation (5).

The only use of this reagent in cement analysis has been reported by Lieber (6). He used indirect procedures in which the precipitated potassium tetraphenylboron is dissolved in acetone and determined titrimetrically.

A direct and simple method has been found for this determination. The sample is decomposed with acid, silica is removed by filtration, and the potassium is precipitated and weighed as the tetraphenylborate.

Reagents

Sodium Tetraphenylboron. J. T. Baker Chemical Company reagent grade was used with no further purification.

Procedure

1. A representative sample that contains approximately ten milligrams of K_2O is weighed exactly and transferred to a small beaker. For most cements an approximately two-gram sample will be about right. For cements of unusual potassium content an adjustment in sample size may be necessary after a preliminary determination.

2. About 20 ml. of water are added and the cement is dispursed thoroughly with a stirring rod.

3. Slightly more than enough concentrated HCl to decompose the sample is added. For a 2-g. sample, 5 ml. of HCl is proper. The acid is added through a funnel while the beaker is covered with a watch glass. When the reaction has subsided the watch glass and the sides of the beaker are washed down with a minimum amount of water and

any undecomposed cement is ground with a flat-base glass rod to facilitate decomposition. Gentle heat can be used also in exceptional cases.

4. The contents of the beaker are cooled if necessary and then filtered through a rapid-filtered paper such as Schleicher and Schnell No. 589 Black Ribbon. The silica residue is washed with water and the filtrate, in a beaker, is diluted to approximately 100 ml.

5. One milliliter of concentrated HCl is added.

6. Approximately 0.25 g. of the sodium tetraphenylboron reagent is dissolved in about 35 ml. of water. This solution is then added to the sample filtrate slowly and with constant stirring.

7. The precipitate of potassium tetraphenylboron is filtered immediately through a weighed, medium-porosity fritted-disc filtering crucible.

8. The precipitate is washed five times with about 2-ml. portions of water. The vacuum is interrupted for the addition of each increment of wash water.

9. The crucible and its contents are then dried for 1-2 hours at 110-120C, cooled in a dessicator, and weighed.

10. The potassium content of the cement is calculated. The conversion factor of $KB(C_6H_5)_4$ to K_2O is 0.1314.

Discussion

The size of the original sample is determined by the weighing error that can be tolerated. The sample size recommended will give an average error of less than 1/4 of 1 per cent.

The precipitation at room temperature seemed to give no difficulties and therefore the solution was not cooled before the

precipitation. Occasional samples did filter very slowly through the fritted-disc crucibles. These samples also gave buff-colored precipitates and high values for potassium. The precipitates of potassium tetraphenylboron should be white after drying and should filter rapidly. Only occasional runs gave the trouble indicated. This difficulty is probably due to decomposition of the reagent for unknown reasons.

The recommended drying times are sufficient but longer drying does no harm. Wendlandt (7) has shown that no decomposition of potassium tetraphenylboron occurs until a temperature of about 300 C is reached.

The total time needed for the determination is about two hours.

Results

The exploratory work on this determination was done on "synthetic-cement" solutions, i.e., solutions containing all the components of decomposed portland cement and known quantities of potassium. In Table I are listed the results of determinations on standard samples of portland cement. The individual results of duplicate runs are given. The standard cements used were from the National Bureau of Standards (NBS) and the Portland Cement Association (PCA).

TABLE I

Sample	Standard Values, %		Tetraphenylboron Method results, %	
	Gravimetric	Flame-Photom		
NBS No. 177	0.57		0.55	0.55
NBS No. 177	0.57		0.56	0.55
NBS No. 177	0.57		0.55	0.55
PCA No. 2	0.24	0.25	0.25	0.25
PCA No. 3	0.67	0.70	0.66	0.68
PCA No. 4	0.14	0.14	0.14	0.14

Conclusions

A rapid, simple, inexpensive, and direct method has been found for the gravimetric determination of potassium in cement using the tetraphenylboron precipitation procedure.

Acknowledgements

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References

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